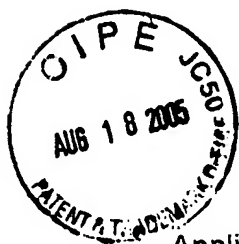


EV633265355



## IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Application Serial No. .... 10/625,166  
 Filing Date ..... July 22, 2003  
 Inventor ..... Janos Fucsko et al.  
 Assignee ..... Micron Technology, Inc.  
 Group Art Unit ..... 2813  
 Examiner ..... Erik J. Kielin  
 Attorney's Docket No. .... MI22-2246  
 Title: Wet Etching Method of Removing Silicon From a Substrate and  
 Method of Forming Trench Isolation

DECLARATION PURSUANT TO 37 C.F.R. § 1.132

To: Commissioner for Patents  
 P. O. Box 1450  
 Alexandria, VA 22313-1450

I, Janos Fucsko, hereby declare as follows:

1. From the Technical University of Budapest, I received the degree B. Sc. In Chemical Engineering in 1979, the degree M. Sc. In Chemical Engineering in 1981, and the degree Doctor Univ. In Analytical Chemistry in 1988.

2. I have been employed by Micron Technology, Inc. since September 2000 as a Process Engineer in Wet Etch Development.

3. I consider myself a person of skill in the wet etching art, and competent to perform approximate calculations of pH of etching solutions within a typical acceptable error range of +/- 0.5 accuracy.

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4. I have reviewed and considered pages 5 and 6 of the Office Action dated April 18, 2005 (hereinafter the "Office Action") in the above captioned patent application where the Patent Examiner makes assumptions and calculations regarding what U.S. Patent No. 5,650,043 discloses at col.5, lines 9-15 regarding an etching solution there disclosed (hereinafter "the etching solution").

5. The Patent Examiner's calculation of a pH of 13.6 for the etching solution in the Office Action is in error. This is due to the calculation error of the  $\text{NH}_4\text{OH}$  concentration, an incorrect assumption of  $\text{NH}_4\text{OH}$  dissociation, and neglecting the contribution of  $\text{NH}_4\text{F}$  to the pH of this buffered etching solution. Rather, I have calculated the pH to be 8.43  $\pm 0.2$ , as is supported by the two page Appendix hereto (hereafter the "Appendix"). The error range is estimated as  $\pm 0.2$  due to typical error sources of neglecting activity coefficients, uncertainty of equilibrium constants, density of solutions before/after mixing, etc. The approximations used for the calculations were for simplification only, are typical, and do not add excessive error to the calculation. Further, this pH calculation was experimentally verified at my direction by the Analytical Laboratory of Micron Technologies, Inc. by mixing together a similar solution, and the similar solution was found to be a good approximation of the pH calculation.

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6. The purpose of the calculations and experiment referred to herein and in the Appendix is to show that the pH calculation in the Office Action was faulty, and not to provide a 100% scientifically accurate method for pH calculation.

7. As the person signing below, I hereby declare that all the statements made herein of my own knowledge are within the generally accepted engineering and scientific practice, and that all the statements made on information and belief are believed to be correct within the acceptable 'engineering error range' and those were made for the illustration of a better/more technical way of problem solving - and not to demonstrate absolute scientific accuracy, and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Inventor:

JANOS FUCSKO

Inventor Signature:

Janos Fucsko

Date:

08/17/05

Residence:

Boise, Idaho

Citizenship:

United States of America

Mailing Address:

6193 South Kelton Place, Boise, ID 83716

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## Appendix

Calculations Pertinent To Example 3 Etching Solution in U.S. Patent 5,650,043,  
Column 5, Lines 8 - 15.

**A. The  $\text{NH}_4\text{OH}$  concentration determined by the Patent Examiner in the Office Action dated April 18, 2005 is in error.**

1. I reasonably conclude that the mixing of a 40%  $\text{NH}_4\text{F}$  and a 28%  $\text{NH}_3$  containing  $\text{NH}_4\text{OH}$  solution is done by weight.
2.  $\text{NH}_4\text{OH}$  weight % can be calculated by adjusting  $\text{NH}_3\%$  with molecular weight ratio,  $M_{\text{NH}_3} = 17$ ,  $M_{\text{NH}_4\text{OH}} = 35$ ,  $\text{NH}_4\text{OH}\% = 35/17 * 28\% = 57.65\%$
3. Molar ratio of  $\text{NH}_4\text{F}$  ( $M_{\text{NH}_4\text{F}} = 37$ ) and  $\text{NH}_4\text{OH}$  ( $M_{\text{NH}_4\text{OH}} = 35$ ) is  
Original 10:1 mixture, assuming a density of 1 g/mL or 1 kg/L:  
 $C_{\text{NH}_4\text{F}} = 10/11 * 400/37 = 9.828 \text{ mol/L} \approx 9.83 \text{ mol/L}$   
 $C_{\text{NH}_4\text{OH}} = 1/11 * 576.5/35 = 1.4974 \text{ mol/L} \approx 1.5 \text{ mol/L}$   
The ratio of the two concentrations:  $= 9.83/1.5 = 6.563$  ( $\text{NH}_4\text{F}$  conc. is therefore 6.563 higher than  $\text{NH}_4\text{OH}$  conc.)  
This ratio will remain during dilution with DI water.

So when  $\text{NH}_4\text{F}$  concentration reached 0.27 M, as stated,  $\text{NH}_4\text{OH}$  concentration should be 6.563 lower, i.e.

$$C_{\text{NH}_4\text{F}} = 0.27 \text{ mol/L}$$

$$C_{\text{NH}_4\text{OH}} = 0.27/6.563 = 0.0411 \text{ mol/L}$$

(Even if volume ratio is used instead of weight, the calculated  $\text{NH}_4\text{OH}$  concentration should still be within 10% of 0.0411 mol/L.)

Calculated dilution ratio ~ 36.4

**B. The pH concentration determined by the Patent Examiner in the Office Action dated April 18, 2005 is in error.**

It is correct that at  $t=25^\circ\text{C}$ ,  $[\text{H}^+] * [\text{OH}^-] = 10^{-14}$

It is incorrect to conclude that  $\text{NH}_4\text{OH}$  completely dissociates in water. Instead,  $\text{NH}_4\text{F}$  is a salt – strong electrolyte, practically fully dissociating, having a degree of dissociation  $\alpha \approx 1$ .

However,  $\text{NH}_4\text{OH}$  is a weak base, dissociating as follows, as reported by page 5-14 of Lange's Handbook Of Chemistry



$$K_{b, \text{NH}_4\text{OH}} = [\text{NH}_4^+] * [\text{OH}^-] / [\text{NH}_4\text{OH}] = 1.75 * 10^{-5} \text{ M (or mol/L)}$$

With that, and knowing that there is at least 0.27 M  $\text{NH}_4^+$  ions in the solution only from  $\text{NH}_4\text{F}$ , the  $\text{NH}_4\text{OH}$  dissociation is pushed back by the excess of  $\text{NH}_4^+$ , so the  $[\text{OH}^-]$  can be calculated as follows:

$$[\text{OH}^-] = 1.75 \times 10^{-5} \text{ M} \cdot [\text{NH}_4\text{OH}] / [\text{NH}_4^+] \approx 1.75 \times 10^{-5} \text{ M} \cdot 0.0411 / 0.27 \text{ M} = 2.667 \times 10^{-6} \text{ M} (*)$$

(So addition of  $2.667 \times 10^{-6} \text{ M}$  to the 0.27 M  $\text{NH}_4^+$  from  $\text{NH}_4\text{F}$  is negligible.)

Therefore, the dissociation degree of  $\text{NH}_4\text{OH}$  is  $\alpha \approx 2.667 \times 10^{-6} \text{ M} / 0.0411 = 6.48 \times 10^{-5}$  (not  $\alpha \approx 1$  as erroneously concluded by the Patent Examiner, and that causes a  $1/6.48 \times 10^{-5} = 15428$  times error in  $[\text{OH}^-]$ , a significant over-estimation).

Accordingly, the true pH can be calculated as:

$$\text{pH} = 14 + \log [\text{OH}^-] = 14 + \log (2.667 \times 10^{-6} \text{ M}) = 8.43 (+/- 0.2)$$

The error range is estimated as +/- 0.2 due to typical error sources of neglecting activity coefficients, uncertainty of equilibrium constants, density of solutions before/after mixing, etc. The approximations used for the calculations were for simplification only, are typical, and do not add excessive error to the calculation. Further, this pH calculation was experimentally verified at my direction by the Analytical Laboratory of Micron Technologies, Inc. by mixing together a similar solution, and the similar solution was found to be a good approximation of the pH calculation.

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